

FORM PTO-1390  
(REV. 11-94)U. S. DEPARTMENT OF COMMERCE  
PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

2988-0695

10/031113

pc

**TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)**INTERNATIONAL APPLICATION NO  
PCT/FR00/01579INTERNATIONAL FILING DATE  
June 8, 2000PRIORITY DATE CLAIMED  
July 16, 1999

## TITLE OF INVENTION

AMINO GUANIDINE BICARBONATE WITH PARTICULAR PROPERTIES AND PROCESS FOR MANUFACTURING IT

## APPLICANT(S) FOR DO/EO/US

Jean-Michel Bossoutrot and Paul Bourdauducq

Applicant herewith submits to the United States Designated/ Elected Office (DO/EO/US) the following items under 35 U.S.C. 371:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
  - a. ☐ is transmitted herewith (required only if not transmitted by the international Bureau).
  - b. ☒ has been transmitted by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
- ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
- ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
  - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☐ have been transmitted by the International Bureaus.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☐ have not been made and will not be made.
- ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 37(c)(3)).
- ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
- ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

**Items 11. to 16. below concern document(s) or information included:**

- ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.  
☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information:
  - International Publication WO 01/05752 A1
  - International Preliminary Examination Report (PCT/IPEA/409)
  - PCT/IB/306

17. ☒ The U.S. National Fee (35 U.S.C. 371(c)(1)) and other fees as follows:

CLAIMS				
(1)FOR	(2)NUMBER FILED	(3)NUMBER EXTRA	(4)RATE	(5)CALCULATIONS
TOTAL CLAIMS	13 -20	0	X \$18.00	\$ 0.00
INDEPENDENT CLAIMS	3 -3	0	X \$84.00	0.00
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$280.00	<input type="checkbox"/>
BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)): <b>CHECK ONE BOX ONLY</b>				
<input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1.482) ..... \$710.00				
<input type="checkbox"/> No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) ..... \$740.00				
<input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$1,040.00				
<input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2) to (4) ..... \$100.00				
<input checked="" type="checkbox"/> Filing with EPO or JPO search report ..... \$890.00				\$ 890.00
Surcharge of \$130.00 for furnishing the National fee or oath or declaration later than 20 30 mos. from the earliest claimed priority date (37 CFR 1.492(e)).				
0			TOTAL OF ABOVE CALCULATIONS	= 890.00
Reduction by 1/2 for filing by small entity, if applicable. Affidavit must be filed also. (Note 37 CFR 1.9, 1.27, 1.28).				- \$ 0.00
			SUBTOTAL	= 890.00
Processing fee of \$130.00 for furnishing the English Translation later than 20 30 mos. from the earliest claimed priority date (37 CFR 1.492(f)).				+
			TOTAL FEES ENCLOSED	\$ 890.00

- a. ☐ A check in the amount of \$\_\_ to cover the above fees is enclosed.
- b. ☒ Please charge Deposit Account No. 16-1150 in the amount of \$890.00 to cover the above fees. A copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 16-1150. A copy of this sheet is enclosed.

18. ☒ Other instructions  
Please calculate the fee after entering the Preliminary Amendment.

19. ☒ All correspondence for this application should be mailed to  
PENNIE & EDMONDS LLP  
1155 Avenue of Americas  
New York, N.Y. 10036-2711

20. ☒ All telephone inquiries should be made to

Charles E. Miller  
NAME

SIGNATURE

24,576  
REGISTRATION NUMBER

January 11, 2002  
DATE



10/031113  
JC13 Rec'd PCT/PTO 11 JAN 2002

**EXPRESS MAIL NO.: EL 477 033 690 US**

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

Application of: Bossoutrot et al.

Group Art Unit: To be assigned

Serial No.: to be assigned

Examiner: To be assigned

Filed: Concurrently filed

Attorney Docket No.: 2988-695

For: AMINO GUANIDINE BICARBONATE  
WITH PARTICULAR PROPERTIES  
AND PROCESS FOR  
MANUFACTURING IT

New York, NY  
January 11, 2001

**PRELIMINARY AMENDMENT**

Assistant Commissioner for Patents  
Washington, D.C. 20231

Sir:

Applicant respectfully requests entry of the following amendment and remarks in to the file of the above identified application.

**IN THE CLAIMS:**

Please cancel claims 1-9.

Please add the following new claims:

10. (New) A process for manufacturing aminoguanidine bicarbonate, comprising combining an aqueous solution of cyanamide and an aqueous solution of hydrazine hydrate to form a reaction medium in the presence of CO<sub>2</sub>, wherein the molar ratio of the cyanamide to the hydrazine used in the process is smaller than 1.

11. (New) The process as claimed in claim 10, wherein the cyanamide/hydrazine molar ratio used is between about 0.8 and about 0.99.

12. (New) The process as claimed in claim 11, wherein the cyanamide/hydrazine molar ratio is between about 0.85 to about 0.95.

13. (New) The process as claimed in claim 10, wherein the pH of the reaction medium is between about 6.5 and about 8.

14. (New) The process as claimed in claim 13, wherein the pH of the reaction medium is between about 7 and about 7.3.

15. (New) The process as claimed in claim 10, wherein the temperature of the reaction medium is between about 35°C and about 70°C.

16. (New) The process as claimed in claim 15, wherein the temperature of the reaction medium is between about 40°C and about 50°C.

17. (New) The process as claimed in claim 10, wherein the pH of the hydrazine hydrate solution is adjusted using CO<sub>2</sub>, and the aqueous cyanamide solution is then introduced.

18. (New) The process as claimed in claim 10, wherein the hydrazine hydrate solution and CO<sub>2</sub> are simultaneously added to the cyanamide solution.

19. (New) The process as claimed in claim 10, wherein the cyanamide solution or of the hydrazine hydrate solution is kept being added to the reaction medium for between about 1 and about 3 hours.

20. (New) A process for manufacturing aminoguanidine bicarbonate, comprising combining an aqueous solution of cyanamide and an aqueous solution of hydrazine hydrate to form a reaction medium in the presence of CO<sub>2</sub>, wherein the molar ratio of the cyanamide to the hydrazine used in the process is between about 0.8 and about 0.99, wherein the pH of the reaction medium is between about 6.5 and about 8, and wherein the temperature of the reaction medium is between about 35°C and about 70°C.

21. (New) A virtually spherical aggregate of amino guanidine bicarbonate crystals with a mean diameter of between about 80 and about 500 µm.

22. (New) The virtually spherical aggregate of amino guanidine bicarbonate crystals of claim 21, wherein the mean diameter is between about 100 and about 250 µm.

**REMARKS**

Claim 1-9 has been canceled. Claims 10-22 have been added.

Support for the new claims can be found in the original specification, e.g., original claims. None of the amendment has narrowed scope of claims. No new matter is believed to be introduced.

No fee, other than that for the application filing fee, is believed due for the filing of this response. Should any fees be required, however, please charge such fees to Pennie & Edmonds LLP Deposit Account No. 16-1150.

Respectfully submitted,

Date: January 11, 2002



Charles E. Miller  
**PENNIE & EDMONDS LLP**  
1155 Avenue of the Americas  
New York, New York 10036-2711  
(212) 790-9090

24,576  
(Reg. No.)

Attorneys For Applicant

3/p<sub>rt</sub>

WO 01/05752

1

PCT/FR00/01579

AMINOGUANIDINE BICARBONATE WITH PARTICULAR PROPERTIES  
AND PROCESS FOR MANUFACTURING IT

The present invention relates to a process  
5 for manufacturing aminoguanidine bicarbonate. The  
invention also relates to an aminoguanidine bicarbonate  
with particular properties.

The manufacture of aminoguanidine bicarbonate  
(AGB) by reacting an aqueous solution of cyanamide with  
10 hydrazine followed by an addition of CO<sub>2</sub> is known. Since  
the placing in contact of cyanamide with hydrazine in  
alkaline medium also leads to the dimerization of the  
cyanamide, it is necessary to use a large excess of  
cyanamide to achieve a suitable yield of aminoguanidine  
15 bicarbonate.

Thus, patent DD 689 191 teaches working with  
a 100% excess of cyanamide (i.e. cyanamide/hydrazine  
molar ratio = 2/1) to obtain a yield (relative to the  
hydrazine used) of 80% of AGB, after reaction for 60  
20 hours. An AGB yield of about 90% may be achieved after  
reaction for 27 hours when concentrated solutions of  
cyanamide and hydrazine are used with a 100% excess of  
cyanamide (DD 730 331).

Since cyanamide is a very expensive product,  
25 attempts to reduce this excess have been the subject of  
much research.

Specifically, patent SU 981 314 discloses a cyanamide/hydrazine molar ratio of between 1.25 and 1.8. It is mentioned that an AGB yield (relative to the hydrazine) of 95% is obtained with a

5 cyanamide/hydrazine molar ratio of 1.8. It also teaches that the yields fall to 90% and 85% for molar ratios of 1.5 and 1.25, respectively.

The same trend has been observed by other authors. Thus, a fall of about 12 points in the yield  
10 was recorded when the cyanamide/hydrazine molar ratio went from 1.2 to 1 (DD 249 009).

The combined literature in this field encourages a person skilled in the art to work with an excess of cyanamide to obtain a yield of aminoguanidine  
15 bicarbonate.

The Applicant Company has developed a process for manufacturing aminoguanidine bicarbonate from cyanamide and hydrazine and has observed, surprisingly, by working with a slight deficit of cyanamide relative  
20 to the stoichiometric amount, yields of AGB that are as high as, or even higher than, those achieved with processes using a large excess of cyanamide.

According to the present invention, the process consists in reacting an aqueous solution of  
25 cyanamide with an aqueous solution of hydrazine hydrate in the presence of  $\text{CO}_2$ , characterized in that the



process is performed with a slight deficit of cyanamide relative to the stoichiometry.

The cyanamide/hydrazine molar ratio used is preferably between 0.80 and 0.99 and advantageously  
5 between 0.85 and 0.95.

The pH of the reaction medium is generally between 6.5 and 8, preferably between 7 and 7.3. The pH may be adjusted by any suitable means and especially using CO<sub>2</sub>.

10 The concentration of the aqueous solutions may vary within a wide range. It is usually preferred to use an aqueous cyanamide solution with a concentration of between 15% and 50% by weight. The hydrazine concentration in the aqueous solution is  
15 advantageously between 15% and 64% by weight.

The temperature of the reaction medium is generally between 35°C and 70°C. A temperature of between 40°C and 50°C gives an aminoguanidine bicarbonate whose structure and specific properties are  
20 commercially very advantageous.

One embodiment consists in adjusting, using CO<sub>2</sub> (carbon dioxide), the pH of the hydrazine hydrate solution to the desired value and then in introducing an aqueous solution of cyanamide once the temperature  
25 of the hydrazine solution has been raised to about a few degrees below the temperature chosen for the reaction.

The pH of the reaction medium is maintained using CO<sub>2</sub> at the desired value during the introduction, or addition, of the cyanamide solution and throughout the reaction.

5 Another embodiment consists in simultaneously adding an aqueous hydrazine hydrate solution and carbon dioxide to an aqueous cyanamide solution initially maintained a few degrees below the temperature chosen for the reaction.

10 Irrespective of the embodiment, the total reaction time is generally between 6 and 15 hours and preferably between 7 and 10 hours. The duration of the addition of cyanamide or of hydrazine hydrate is generally between 1 and 3 hours and preferably in the  
15 region of 2 hours.

After the reaction, the reaction medium is cooled to room temperature and the aminoguanidine bicarbonate thus obtained is spin-filtered or filtered and optionally dried.

20 With the process according to the invention, yields of greater than 90% and preferably greater than 95% are obtained with a purity of greater than 99%, or even greater than 99.5%.

A subject of the present invention is also an  
25 aminoguanidine bicarbonate with a particular structure and particular specific properties. It is characterized by a virtually spherical crystal aggregate with a mean

diameter of between 80 and 500  $\mu\text{m}$ . The aggregate preferably has a mean diameter of between 100 and 250  $\mu\text{m}$ , the mean diameter being determined by laser granulometry.

5           The aminoguanidine bicarbonate according to the invention also has the advantage of being easy to separate out from the reaction medium by any known means, for example by filtering or spin-filtering and drying, thus being distinguished from platelet  
10 crystals.

#### EXPERIMENTAL SECTION

##### Example 1

110.9 g of hydrazine hydrate with a purity of 99.2% (2.2 mol) and 300 g of demineralized water are  
15 placed in a one liter reactor at room temperature. The pH of the aqueous solution is in the region of 11. Carbon dioxide is then bubbled into the aqueous solution for about 1 hour, which represents 58 g or 1.3 mol of  $\text{CO}_2$ , until a pH in the region of 7 is  
20 obtained, while maintaining the temperature of the solution at about 40°C.

171.4 g of an aqueous 49% cyanamide solution (2 mol) are then added over about 2 hours, while continuing the addition of  $\text{CO}_2$  so as to maintain the pH  
25 of the reaction medium in the region of 7. During the addition, the temperature of the medium is raised to 45°C and the medium is maintained at this temperature

for 8 hours with adjustment of the pH to a value in the region of 7 by small additions of CO<sub>2</sub>.

The total amount of CO<sub>2</sub> added is 104 g, i.e. 2.36 mol.

5           At the end of the reaction, the reaction medium is allowed to cool to room temperature and the AGB crystals are then filtered off and washed with 250 ml of water and finally dried under vacuum at a temperature of between 35°C and 40°C.

10           After drying, 260 g of crystals with a purity of 99.7%, determined by assaying with perchloric acid, are obtained. The crude yield relative to the cyanamide is 95.6%.

          The crystals obtained are in the form of  
15 virtually spherical aggregates (photograph No. 1 by scanning electron microscopy).

#### Example 2

          The process is performed as described in Example 1, except that the aqueous hydrazine hydrate  
20 solution is maintained at 55°C instead of 40°C and that, during the addition of the cyanamide, the reaction medium is brought to 65°C and is maintained at this temperature for 4 hours.

          After drying, 261.1 g of crystals in the form  
25 of platelets (photograph No. 2) are obtained <sup>with</sup> ~~in~~ a purity of 99.6%. The crude yield relative to the cyanamide is 96%.

Example 3

Example 1 is extrapolated to the industrial scale, using a 15 m<sup>3</sup> reactor.

After spin-filtering for 20 minutes, the aggregates contain a moisture content of only 7%. At the end of the spin-filtering, the aggregates are virtually spherical of the type in Example 1, with a narrow particle size distribution free of fine particles, of less than 40 µm in diameter.

Example 4

Example 2 is repeated on the industrial scale, using a 15 m<sup>3</sup> reactor.

After spin-filtering for 3 hours, the platelets contain a moisture content of 20% and, at the end of the spin-filtering, the platelets have a mean diameter of 70 µm with a very broad particle size distribution with 20% of the population of particles having a diameter of less than 20 µm.

Example 5

The procedure described in Example 1 is repeated, except that the duration of addition of the cyanamide is 5 hours instead of 2 hours and the duration of the reaction after the addition is reduced from 8 to 5 hours.

The yield and also the purity of the AGB crystals obtained are similar to those of Example 1. However, the crystals are rather in the form of

platelets (photograph No. 3) and the spin-filtering time is longer.

Example 6

171.4 g of an aqueous 49% cyanamide solution  
5 (2 mol) and 300 g of water are placed in a one liter reactor at room temperature. The pH of the resulting solution is in the region of 5. The solution is then brought to 40°C, after which 110.9 g of 99.2% hydrazine hydrate (2.2 mol) and 75 g (1.7 mol) of CO<sub>2</sub>, to maintain  
10 the pH at about 7, are simultaneously added over 2 hours. The reaction medium is then maintained at 45°C for 8 hours with a small addition of CO<sub>2</sub> to adjust the pH to about 7. The total amount of CO<sub>2</sub> added is 94.5 g (2.15 mol).

15 The reaction medium is then allowed to cool to room temperature and the AGB is filtered off and washed with 250 ml of water. Finally, it is dried under vacuum at a temperature of between 35°C and 40°C.

After drying, 259 g of AGB aggregates similar  
20 to those of Example 1, with a purity of 99.6%, are obtained.

The crude yield of aminoguanidine bicarbonate is 95.2% relative to the cyanamide.

## CLAIMS

1. A process for manufacturing  
5 aminoguanidine bicarbonate from an aqueous solution of  
cyanamide and an aqueous solution of hydrazine hydrate  
in the presence of  $\text{CO}_2$ , characterized in that the  
process is performed with a slight deficit of cyanamide  
relative to the stoichiometry.
- 10 2. The process as claimed in claim 1,  
characterized in that the cyanamide/hydrazine molar  
ratio used is between 0.8 and 0.99.
3. The process as claimed in claim 2,  
characterized in that the cyanamide/hydrazine molar  
15 ratio is between 0.85 and 0.95.
4. The process as claimed in one of claims  
1 to 3, characterized in that the pH of the reaction  
medium is between 6.5 and 8 and preferably between 7  
and 7.3.
- 20 5. The process as claimed in one of claims  
1 to 4, characterized in that the temperature of the  
reaction medium is between  $35^\circ\text{C}$  and  $70^\circ\text{C}$  and preferably  
between  $40^\circ\text{C}$  and  $50^\circ\text{C}$ .
6. The process as claimed in one of claims  
25 1 to 5, characterized in that the pH of the hydrazine  
hydrate solution is adjusted using  $\text{CO}_2$ , and the aqueous  
cyanamide solution is then introduced.

7. The process as claimed in one of claims 1 to 5, characterized in that an aqueous hydrazine hydrate solution and carbon dioxide are simultaneously added to an aqueous cyanamide solution.

5 8. The process as claimed in either of claims 6 and 7, characterized in that the duration of the addition of cyanamide or of hydrazine hydrate is between 1 and 3 hours.

10 9. A virtually spherical aggregate of amino guanidine bicarbonate crystals with a mean diameter of between 80 and 500  $\mu\text{m}$  and preferably between 100 and 250  $\mu\text{m}$ .

206240-ETFEOT



(19) World Intellectual Property Organization

International Bureau

WIPO

(43) International publication date

25 January 2001 (25.01.2001)

PCT

(10) International publication number

WO 01/05752 A1

(51) International patent classification<sup>7</sup>:

C07C 277/02, 279/02

(21) International application number: PCT/FR00/01579

(22) International filing date: 8 June 2000 (08.06.2000)

(25) Language of filing: French

(26) Language of publication: French

(30) Data relating to the priority:

99/09,257 16 July 1999 (16.07.1999)

FR

(71) Applicant (for all designated States except US):

ATOFINA [FR/FR]; 4/8, cours Michelet, F-92800  
Puteaux (FR)

(72) Inventors; and

(75) Inventors/Applicants (US only): BOSSOUTROT, Jean-  
Michel [FR/FR]; 11, avenue de Gadagne, F-69230 Saint-Genis  
Laval (FR). BOURDAUDUCQ, Paul [FR/FR]; 8, rue des  
Anémones, F-69630 Chaponost (FR).

(74) Representative: DANG, Doris; Elf Atochem S.A.,  
DCRD/DPI, Cours Michelet, La Défense 10, F-92091  
Paris la Défense Cedex (FR).

(81) Designated states (*national*): AE, AL, AM, AT, AU,  
AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU,  
CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM,  
HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ,  
LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK,  
MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE,  
SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US,  
UZ, VN, YU, ZA, ZW.

(84) Designated states (*regional*): ARIPO Patent (GH, GM,  
KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW),  
Eurasian Patent (AM, AZ, BY, KG, KZ, MD, RU, TJ,  
TM), European Patent (AT, BE, CH, CY, DE, DK, ES,  
FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). OAPI  
Patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML,  
MR, NE, SN, TD, TG).

Published:

- With the International Search Report.

*For an explanation of the two-letter codes and the other  
abbreviations, reference is made to the explanations  
("Guidance Notes on Codes and Abbreviations") at the  
beginning of each regular edition of the PCT Gazette.*

As printed

(54) Title: AMINOGUANIDINE BICARBONATE WITH PARTICULAR PROPERTIES AND METHOD FOR MAKING SAME

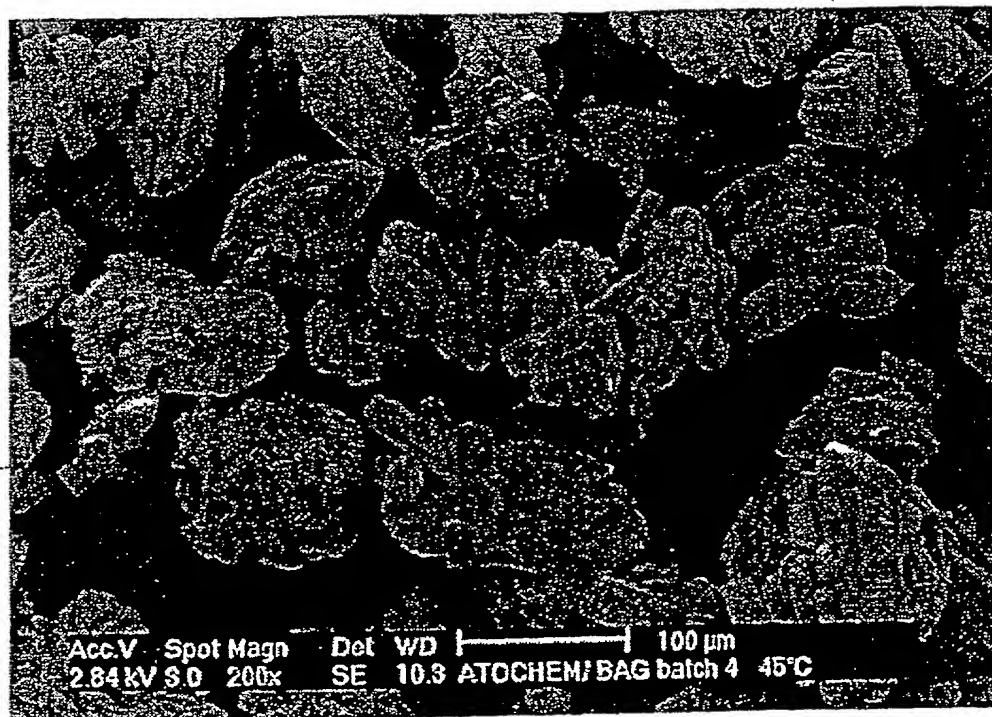
(54) Titre: BICARBONATE D'AMINOGUANIDINE DE PROPRIETES PARTICULIERES ET SON PROCEDE DE FABRICATION

(57) Abstract: The invention concerns a method for making aminoguanidine bicarbonate from an aqueous solution of cyanamide and an aqueous solution of hydrazine hydrate in the presence of CO<sub>2</sub>. The invention is characterised in that it consists in proceeding with an amount of cyanide slightly higher than the stoichiometric quantity. The invention also concerns quasi-spherical agglomerates of aminoguanidine bicarbonate crystals.

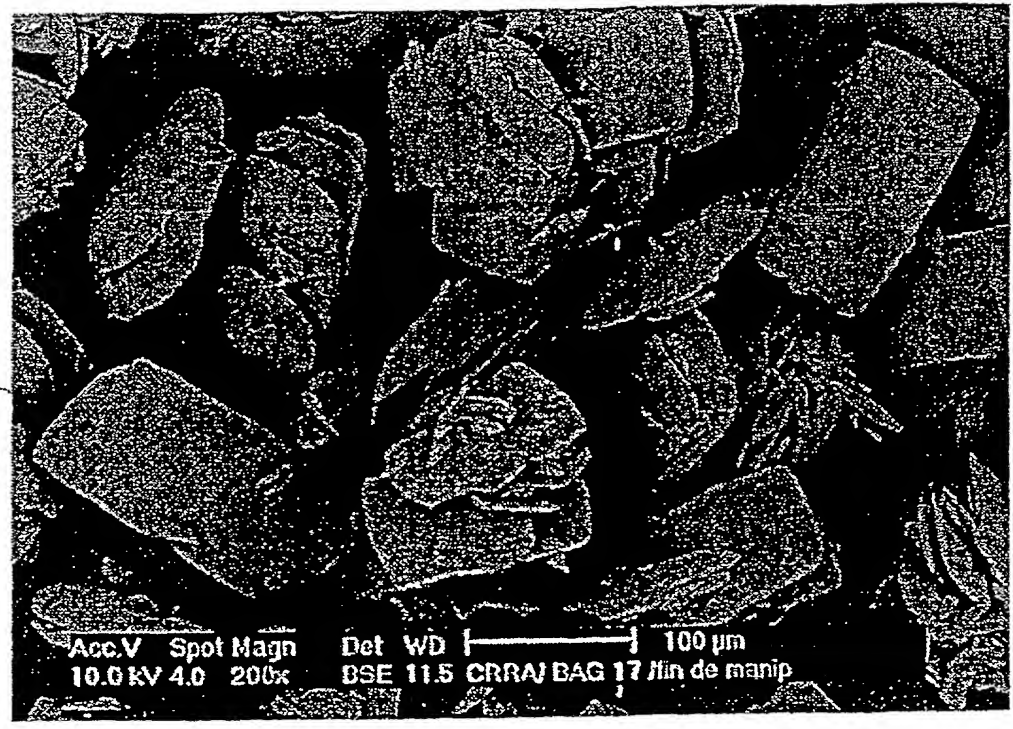
(57) Abrégé: La présente invention concerne un procédé de fabrication de bicarbonate d'aminoguanidine à partir d'une solution aqueuse de cyanamide et d'une solution aqueuse d'hydrate d'hydrazine en présence de CO<sub>2</sub> caractérisé en ce que l'on opère avec un léger défaut en cyanamide par rapport à la stoechiométrie. Elle a également pour objet des agglomérats quasi sphériques de cristaux de bicarbonate d'aminoguanidine.

WO 01/05752 A1

1/3



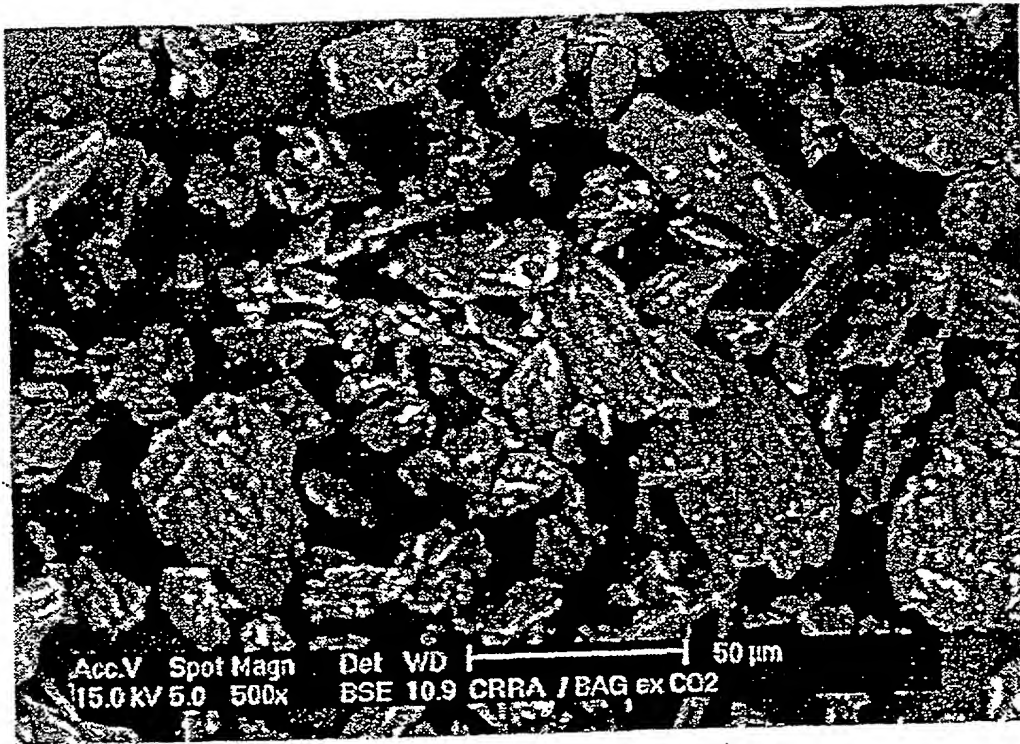
PHOTOGRAPH N° 1/3



PHOTOGRAPH N° 2/3

206240" ETTEDT

3/3



PHOTOGRAPH N° 3/3



## DECLARATION FOR NON-PROVISIONAL PATENT APPLICATION\*

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below at 201 et seq. beneath my name.

I believe I am the original, first and sole inventor if only one name is listed at 201 below, or an original, first and joint inventor if plural names are listed at 201 et seq. below, of the subject matter which is claimed and for which a patent is sought on the invention entitled

## AMINO GUANIDINE BICARBONATE WITH PARTICULAR PROPERTIES AND PROCESS FOR MANUFACTURING IT

and for which a patent application:

☐ is attached hereto and includes amendment(s) filed on (if applicable)

☒ was filed in the United States on as Application No. 10/031,113

with amendment(s) filed on January 11, 2002

☒ was filed as PCT international Application No. PCT/FR00/01579 on June 8, 2000 and was amended under PCT Article 19 on (if applicable)

I hereby state that I have reviewed and understand the contents of the above identified application, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119(a)-(d) of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

EARLIEST FOREIGN APPLICATION(S), IF ANY, FILED PRIOR TO THE FILING DATE OF THE APPLICATION			
APPLICATION NUMBER	COUNTRY	DATE OF FILING (day, month, year)	PRIORITY CLAIMED
99/09257	France	July 16, 1999	YES <input checked="" type="checkbox"/> NO <input type="checkbox"/>
			YES <input type="checkbox"/> NO <input type="checkbox"/>
			YES <input type="checkbox"/> NO <input type="checkbox"/>

I hereby claim the benefit under Title 35, United States Code, §119(e) of any United States provisional application(s) listed below.

PROVISIONAL APPLICATION NUMBER	FILING DATE

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code §112, I acknowledge the duty to disclose information known to me which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

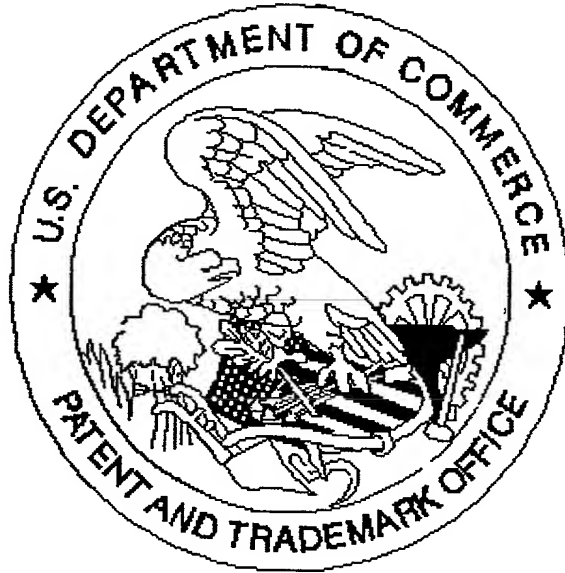
NON-PROVISIONAL APPLICATION SERIAL NO.	FILING DATE	STATUS		
		PATENTED	PENDING	ABANDONED

\* for use only when the application is assigned to a company, partnership or other organization.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

201	FULL NAME OF INVENTOR	LAST NAME Bossoutrot	FIRST NAME Jean-Michel	MIDDLE NAME	
	RESIDENCE & CITIZENSHIP	CITY Chaponost FRX	STATE OR FOREIGN COUNTRY France	COUNTRY OF CITIZENSHIP France	
	POST OFFICE ADDRESS	STREET 8 Chemin de Traine-Fesses	CITY Chaponost	STATE OR COUNTRY France	ZIP CODE F-69630
	SIGNATURE OF INVENTOR 201			DATE April 24, 2002	
202	FULL NAME OF INVENTOR	LAST NAME Bourdauducq	FIRST NAME Paul	MIDDLE NAME	
	RESIDENCE & CITIZENSHIP	CITY Chaponost FRX	STATE OR FOREIGN COUNTRY France	COUNTRY OF CITIZENSHIP France	
	POST OFFICE ADDRESS	STREET 8, rue des Anemones	CITY Chaponost	STATE OR COUNTRY France	ZIP CODE F-69630
	SIGNATURE OF INVENTOR 202			DATE April 24, 2002	
203	FULL NAME OF INVENTOR	LAST NAME	FIRST NAME	MIDDLE NAME	
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP	
	POST OFFICE ADDRESS	STREET	CITY	STATE OR COUNTRY	ZIP CODE
	SIGNATURE OF INVENTOR 203			DATE	
204	FULL NAME OF INVENTOR	LAST NAME	FIRST NAME	MIDDLE NAME	
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP	
	POST OFFICE ADDRESS	STREET	CITY	STATE OR COUNTRY	ZIP CODE
	SIGNATURE OF INVENTOR 204			DATE	
205	FULL NAME OF INVENTOR	LAST NAME	FIRST NAME	MIDDLE NAME	
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP	
	POST OFFICE ADDRESS	STREET	CITY	STATE OR COUNTRY	ZIP CODE
	SIGNATURE OF INVENTOR 205			DATE	

United States Patent & Trademark Office  
Office of Initial Patent Examination -- Scanning Division



Application deficiencies found during scanning:

☐ Page(s) \_\_\_\_\_ of \_\_\_\_\_ were not present  
for scanning. (Document title)

☐ Page(s) \_\_\_\_\_ of \_\_\_\_\_ were not present  
for scanning. (Document title)

☒ Scanned copy is best available. Drawings are too dark